

Magnetic studies of transuranium compounds

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Abstract

The magnetic properties of transuranium compounds are of special interest since their magnetic ions are more localized than those in uranium compounds. Various isostructural classes of compounds do exist such as the NaCl structure compounds, the Laves phases, and compounds of the anti-Cu₂Sb and AuCu₃ structure. It is shown that the availability of single crystals, more powerful experimental techniques and advances in theory have yielded important progress in the last ten years.

1. Introduction

The magnetic properties of the well-localized 4f electrons in rare earths and their compounds became of interest around 1950. The 5f electrons of the actinides are of greater radial extension and an overlap of 5f functions and hybridization with outer electrons leading to band magnetism is more probable than in the case of 4f electrons. Overlap and hybridization are readily encountered in uranium compounds. The 5f electrons should be more localized in heavier actinides. This is the main reason for the interest in transuranium compounds where one hopes to find a simpler kind of magnetism than in uranium compounds. Among novel compounds let us mention the curium pnictides [1, 2]. Using a newly developed superconducting quantum interference device (SQUID) magnetometer, Nave *et al.* [2] were able to measure microgram quantities of CmP and CmSb. The new technology allowed an accurate determination of the temperature dependence of the magnetic susceptibility of CfN, CfAs and CfSb [3]. CfAs and CfSb are antiferromagnets: whether CfN is a ferromagnet as claimed by these researchers needs some further proof.

We shall put some emphasis on cubic systems which are certainly best adapted to microscopic analysis of magnetic properties especially if there is only one magnetic site (the Laves phases AnX₂ with X ≡ Co, Ni, Fe are examples of cubic substances with two magnetic sites, namely the moment of the actinide and the moment of the 3d element). Almost all compounds of Np and Pu have been investigated in the period 1970–1978 mostly at Argonne National Laboratories. Accounts of this tremendous and beautiful work were presented by

Lam and Aldred [4]. Some more recent summaries were given by Fournier and Troč [5].

2. Single crystals

Today we know that the magnetic properties of all actinide compounds (S state ions, *i.e.* Cm³⁺ ions excepted) are highly anisotropic. The moment of a single ion is anisotropic, the response to an applied field of an uncoupled ion is anisotropic and exchange forces as well are anisotropic.

Neutron diffraction experiments can determine readily the moment direction in antiferromagnets even if they are performed on powder samples. For ferromagnetic samples this is normally not possible. However, in a very elaborate way Aldred *et al.* [6] have succeeded in determining the moment direction in NpFe₂ (powder samples) by comparison of polarized neutron results with results obtained on a single crystal of US (which was available at that time, 1975) for which the moment direction is known, namely the $\langle 111 \rangle$ axis.

Today single crystals of transuranium compounds are available for some of the Laves phases, for mononpnictides and monochalcogenides of Pu, Np and Am, some dipnctides and some heavy fermion compounds.

As an example of how successful experiments on even very small crystals can be, let us remember neutron diffraction and Mössbauer studies of a 0.3 mm³ NpAs₂ crystal. Several researchers [7–11] have succeeded in determining the form factor, type of order and the moment direction, the valence of Np, the magnetization and some crystal field parameters.

In addition to ensuring meaningful magnetization measurements single crystals offer other advantages:

stoichiometry is in most cases well defined, micrographic control for single-phase material is possible, and much more sensitive than X-ray patterns, oxidation is less of a problem than in powder samples and surface experiments (optical, X-ray) can be performed.

3. Advanced experiments

Progress is threefold: new techniques have been introduced (SQUID magnetometers, muon spectroscopy, X-ray resonant scattering), neutrons of higher flux are available, and, last but not least, through advances in theory the interpretation of results is more complete.

3.1. Susceptibility measurements

Microgram samples have been measured; samples of 10–20 mg are the rule. The susceptibility behaviour of localized ions could be divided into four different temperature regions: below the ordering temperature (anisotropy of ordered state), just above the ordering temperature (crystal electric field (CEF) effects), intermediate temperature (Curie–Weiss behaviour) and high temperature region (Van Vleck paramagnetism). Furthermore, one often observes a temperature-independent susceptibility, said to be an enhanced Pauli paramagnetism, Van Vleck paramagnetism of crystal field states, or just yet unexplained.

The case of NpP (depicted in Fig. 1) shows how susceptibility measurements can confirm and complete X-ray and neutron results. Clearly recognizable are the Néel temperature T_N , a structural change at T_S and finally a gradual getting in of a ferrimagnetic component below about 30 K. Approaching T_N on cooling down, the inverse susceptibility curve flattens out owing to crystal field splitting of the $5f^4$ ground state.

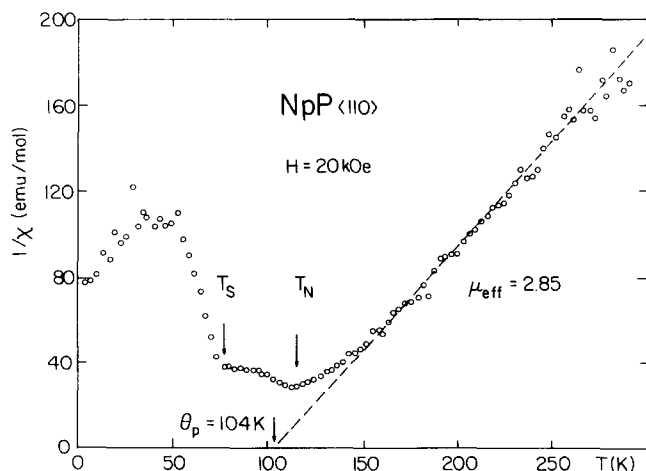


Fig. 1. Inverse magnetic susceptibility vs. temperature of NpP.

Above the ordering temperature, but still in the low temperature region, the influence of the crystal field splitting of the $5f$ ground state of the actinide ions manifests itself. This influence is frequently mentioned but very few exact calculations are available. The influence of the CEF on the susceptibility behaviour causing a flattening out of the inverse susceptibility curve at low temperature (lowest level singlet Γ_1) has been observed for diluted UTe [12]. In contrast, the inverse susceptibility curve of CeP (lowest level doublet Γ_7) turns downwards at low temperature [13]. Only by knowing explicitly the crystal field splitting, *i.e.* the sequence of the terms and the energy differences, can one give a convincing explanation of the susceptibility behaviour. Such calculations were done for the susceptibility of AmO₂ by Karraker [14].

Above the “crystal field region”, *i.e.* at intermediate temperatures, one usually observes Curie–Weiss behaviour. The effective magnetic moments are close to the free-ion value based on Russell–Saunders coupling (intermediate coupling is often considered as well). By extrapolation of the linear Curie–Weiss curve we can determine a paramagnetic Curie temperature θ_{para} . If the antiferromagnetic structure below the ordering temperature T_N is known and if we are sure that exchange forces are isotropic (which is the case for S state ions) we even can calculate exchange forces between nearest and next-nearest neighbours. For anisotropic exchange such calculations make no sense.

At high temperatures normal Van Vleck paramagnetism can become apparent, causing a flattening out of the Curie–Weiss law.

Often non-linearity of the Curie–Weiss curve is “corrected” by applying a modified Curie–Weiss law, namely $\chi = \chi_{cw} + \chi_0$. Although all non- $5f$ electrons together create a certain χ_0 some caveats are nevertheless recommended. Quite often χ_0 values as measured on powder samples decrease by an order of magnitude when measured on a clean single crystal. As an example one may compare powder measurements on UN by Raphael and de Novion [15] with single-crystal measurements by Du Plessis and Van Doorn [16]. On the contrary, Brodsky *et al.* [17] mention that their magnetic measurements on AnRe₂ compounds could be explained either by a modified Curie–Weiss law or by a crystal field effect. Usually no explanation for a temperature-independent paramagnetic contribution to the susceptibility of actinide compounds is given. High temperature measurements are scarce. Raphael and Lallement [18] measured PuO₂ up to 1000 K. No temperature dependence is visible; researchers offer plausible explanations on basis of Van Vleck paramagnetism between crystal field levels.

Enhanced Pauli paramagnetism is another frequently mentioned possibility for the explanation of temper-

ature-independent paramagnetism. The enhancement factors do not seem unreasonable, given that many of the actinide compounds exhibit electric properties which are significant for high effective electron masses.

Wachter *et al.* [19] explain the temperature-independent paramagnetism of the Pu chalcogenides by a mixed-valence state. Thus, in PuTe the Pu ion should have a valence of 2.75. Measurements of mixed crystals of $\text{PuS}_{1-x}\text{Te}_x$ [20] reveal a progressive increase with x of the χ_0 value (which is low for trivalent Pu in PuSb) to the susceptibility values as observed in PuTe.

3.2. Magnetization

Today magnetization measurements are routinely possible between 1.5 K and 300 K in fields usually up to 100 kOe, and in rare cases up to 300 kOe. Since magnetic properties of actinide compounds are as a rule highly anisotropic only measurements on single crystals make sense. Often very high fields are necessary to overcome coercive forces in order to measure a complete hysteresis loop. On the contrary, in induced-moment systems, the magnetic moment in high fields may be considerably different from the zero-field ionic moment. In this case magnetization measurements are not the first choice for the determination of ordered moments: Mössbauer or neutron diffraction experiments are better qualified.

Instructive examples of compounds with extremely high anisotropy are UPd_2Si_2 [21] and NpRu_2Si_2 [22]. Along the a axis their magnetization up to 300 kOe is very small whereas along the c axis a saturation value of about 1 μB is reached in much smaller fields.

Magnetization measurements on an NpCo_2 single crystal [23] allow in conjunction with neutron diffraction data a detailed analysis of the Np and Co moments in this interesting system.

Single crystals of all monochalcogenides and mononitrides of U, Np and Pu are today readily available (nitrides excepted). A summary of all the measurements is given by Vogt and Mattenberger [24]. We concentrate on some examples. The hysteresis loop of (cubic) NpP is extremely large [25]: 40 kOe are needed to induce the full moment even along the easy direction. The same behaviour is found in NpAs [25]. Anisotropy remains up to 100 kOe. Moments along the off-easy axes are merely the projections of the $\langle 100 \rangle$ easy-axis moment. On the basis of magnetization measurements alone ($M(H)$ at a multitude of temperatures and $M(T)$ in various applied fields) a tentative magnetic phase diagram was elaborated for NpP [25]. A confirmation by neutron experiments as in the case of NpAs [25] is still lacking.

Very interesting anisotropies were found in the magnetization of mixed crystals $\text{NpSb}_x\text{Te}_{1-x}$ and $\text{NpAs}_x\text{Se}_{1-x}$ [26]. The magnetic axis has to change from $\langle 100 \rangle$

(pnictide) to $\langle 111 \rangle$ (chalcogenide). As a consequence a $\langle 110 \rangle$ easy axis is expected at intermediate concentrations as was confirmed by magnetization measurements.

3.3. Neutron scattering

No analysis of magnetization curves is possible as long as the spin structure and the neutron ordered moment are unknown. Spin structures can be determined by inelastic neutron diffraction. Summaries of latest results are given by Lander [27]. Determinations of magnetic structures on powdered or polycrystalline samples are to some extent possible.

The availability of single crystals brought further progress since multi- k structures in actinides were detected. Rossat-Mignod *et al.* [28] found the first multi- k structure in an actinide compound. A summary of multi- k structures is given by Rossat-Mignod [29]. Besides the classical single-, double- and triple- k structures, which are encountered in almost all actinide compounds, it was found more recently that the Fourier components of the magnetization vector exist not only independently of each other but coexistence of ferro- and antiferromagnetic arrangements is possible. The three Fourier components need not be of the same magnitude.

We can only mention some examples of many experiments. The complete magnetic phase diagram for PuSb has been established. As a function of temperature, just below the ordering point, this phase diagram is complicated: there is no direct transition from paramagnetism to ferromagnetism as a result of going to lower temperatures [30].

NpAs is another often-studied example. Magnetization, Mössbauer and X-ray distortion experiments were not able to give a complete magnetic phase diagram even though many details were revealed. By combining magnetization measurements and neutron diffraction data, both performed on single crystals, a complete phase diagram was finally composed by Burlet and coworkers [27, 30].

By adding 5% of NpSe to NpAs we obtain a compound with a very interesting spin structure with a magnetically observed easy axis along $\langle 110 \rangle$. The spin structure is the result of the superposition of two antiferromagnetically ordered Fourier components along two cubic axes with one ferromagnetically ordered component along the third cubic axis [27].

Polarized neutron studies provide a further insight into the ionic properties of magnetic compounds which is much more detailed than the best structure analysis and especially valuable for theoretical interpretations.

Let us just mention as one example the study of the induced magnetic form factor on an NpO_2 single crystal [31] which is indicative for the power of this experimental technique.

3.4. Mössbauer spectroscopy

Mössbauer spectroscopy is a very valuable tool to study the magnetic properties of actinide compounds. Only small quantities of polycrystalline material are needed, which does not even have to be too pure. External pressure or external fields can relatively easily be integrated into the experimental set-up. Mössbauer experiments measure magnetic moments in zero external field. This is very important, since, as it is necessary for magnetization measurements, the application of an external field might change the ionic moment. In all Np compounds the hyperfine field depends linearly on the ordered (neutron) moment. This linear Dunlap–Lander relation allows the determination of magnetic moments by Mössbauer experiments. The isomer shift gives unambiguous information on the valency of the actinide ion.

A summary of Mössbauer results up to the year 1985 is given by Dunlap and coworkers [32, 33]. Most newer results were obtained on neptunium compounds which are ideally suited for Mössbauer experiments. Intermetallics under investigation were NpRu₂ [34] and NpPd₂Al₃ [35] and cubic NpX₃ (X ≡ Ga, In, Ge, Sn) [36]. In the cubic systems information on the crystal field splitting could be obtained.

In NpCo₂ [37, 38] Mössbauer spectroscopy was a valuable complement allowing the verification of results obtained by neutron and magnetization measurements. Compounds of the NaCl-type structure which were investigated are NpSb [39] and NpSb–NpTe mixed crystals [26]. A mixing of the 5f electron states with the p states of the anion was evidenced. For NpS, NpSe [40] and NpBi [41] Mössbauer experiments were the only reliable possibility to determine magnetic properties. Magnetization measurements failed to do so owing to insufficient sample quality.

In the last four years compounds with heavy electrons have become of great interest. A summary of Mössbauer experiments on such Np compounds is given by Kalvius *et al.* [42]. Especially interesting are NpRh₂Si₂ [43] and NpBe₁₃ [44, 45].

Experiments in applied external fields are reported for NpO₂ [46] and Gal *et al.* [47] report on results obtained on Np intermetallics in the paramagnetic state. The dominant contribution to the magnetic moment stems from the orbital moment. Mössbauer studies offer the unique possibility to apply very high pressure. Such experiments have been done on NpAs [48] where two different ordered moments were found and on NpSn₃ [49] where the coexistence of an itinerant moment at the Sn site with a local moment at the Np site could be evidenced.

Of all the plutonium compounds only the antimonides lend themselves to Mössbauer studies using ¹²¹Sb as

a source. Results were obtained for PuSb–PuTe mixed crystals [50] and for pure and diluted PuSb [51].

4. Theory

Results up to the year 1985 have been summarized by Cooper *et al.* [52] and further progress will be reviewed by Cooper in the near future.

We have already mentioned the non-integer number of f electrons observed in PuTe [19], and the determination of the valence of the An ion remains a problem. X-ray, Mössbauer or susceptibility measurements cannot always give a decisive answer. The interatomic spacing, leading to direct overlap of the f electron functions, is certainly an important even though not always a crucial factor. Exceptions to the Hill rule are known today. The theory of crystal field splitting, especially in cubic lattices, is well developed but the magnitude of the overall splitting has to be determined by suitable experiments. Such experiments are not yet possible for Pu or Np components. Isotropic exchange has its roots in the Heisenberg mechanism, in superexchange or in Ruderman–Kittel–Kasuya–Yoshida interactions.

Hybridization leads to anisotropic exchange and moment reduction. Almost all theory is thus concerned with hybridization. The discovery of heavy electron systems had a further impact on theory [53]. Today theory, as used by Cooper and collaborators, is advanced to a level which permits *ab initio* calculations of exchange forces, ordered moments and transitions to the heavy fermion state [54–57].

Certainly our understanding is not yet complete but progress in theory within the last eight years is very admirable.

5. Conclusions

Today, for a great variety of transuranium compounds, the general features of magnetic behaviour are known, mostly on the basis of susceptibility measurements. Detailed analyses of magnetic properties, *i.e.* knowledge of the spin structure, the magnitude of the ordered moment and the complete magnetic phase diagram, are not as frequently encountered. They usually require neutron diffraction, Mössbauer and magnetization measurements carried out on a single crystal. Single crystals, especially large crystals, are still scarce. Mössbauer experiments are readily possible only for Np compounds. Further progress will be linked to the availability of single crystals. New techniques, such as muon spectroscopy or synchrotron radiation experiments, may yield new results. Finally, new compounds with unexpected features might be found.

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